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by

W. J. Bock, A. J. Rostocki

FOREIGN TECHNOLOGY DIVISION

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HOW CAN ONE MILLION ATM. BE OBTAINED?

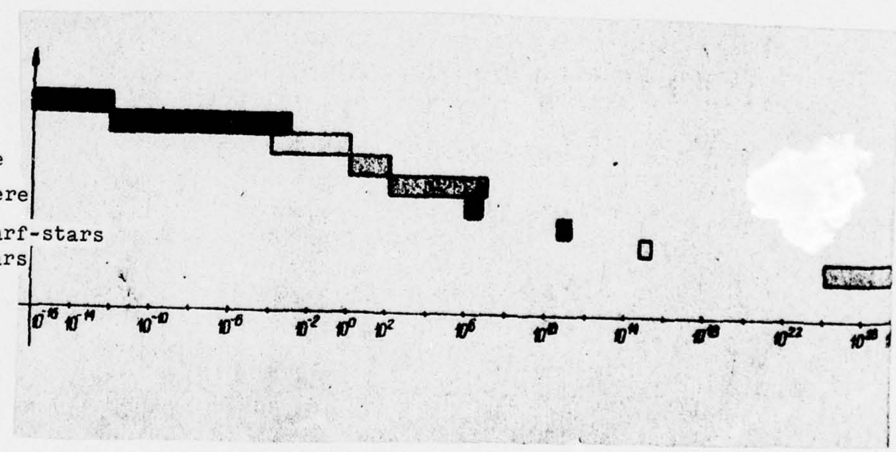
W. J. Bock, A. J. Rostocki

Low and high pressure, to people not familiar with technology, is chiefly associated with a vist to the doctor, but at best, with weather conditions, with the proverbial lower or higher barometric pressure.

Meanwhile under terrestrial conditions, not speaking now of the universe, not many physical quantities are characterized by such a vast range of change, as pressure is. Starting from a high vacuum in circum-earth space, equalling about 10^{-14} Tr, through a narrow pressure range in which life can exist, we come to a pressure value of 4.10^{6} atm. governing the interior of the Earth. (Fig. 1).

Fig. 1 Pressure Scale

cosmic vacuum
high vacuum
industrial vacuum
pressure comp. w/life
high pressure
pressure in bathysphere
pressure inside sun
pressure in white dwarf-stars
pressure inside pulsars



In the eternal drive to know the world's realities which surround us all, man, wanting to expand his extremely limited sensual methods and cognitive capabilities, first of all, had to learn artificially (in a laboratory) to produce and measure certain conditions existing independently in nature, but impossible to investigate by direct contact. Just such conditions are a high vacuum, high pressure, low and high temperature, strong magnetic fields, generally designated in physics as extreme states. It is just that production of such conditions and the execution in those (conditions) of the most varied investigations which, in the largest compendium, is the modus vivendi of contemporary experimental physics.

Spectacular expeditions into the Cosmos brought about the headlong development of electronics and its intervention into practically every field of our life from one side and another, so that in the domain of pressures we are familiar, in fact, with a vacuum. The vacuum, as a structural element of certain devices (electronic tubes, radiations, converters, and the like), as an investigative parameter (observations on the course of various physical-chemical processes under high vacuum conditions) or also as a technological parameter in industry (the production of semi-conductor and thin-layered elements) is known to us from everyday life and we can fancy it for ourselves easily enough. Meanwhile, expeditions into the earth's depths do

not await us for the time being, and so the fund of information on the theme of high pressures is in society considerably smaller, although there can be found many examples of their uses, sometimes in direct, but most often in indirect form, rarely appearing before our eyes in daily life.

Here then, we propose the conclusions of a somewhat more intimate acquaintance with high pressures and the acquaintance with the problems connected with their production. We will concern ourselves first of all, with hydrostatic pressure. The term "static" means in this case that such pressure can maintain a constant value for a long time--from several minutes to several weeks, depending on industrial or even scientific needs.

There are several ways of generating high hydrostatic pressures. Thus, we can produce such pressures using, e. g. , a thermal expansion method--depending on the heating, in a closed high pressure vessel, of bodies having a large coefficient of thermal expansion and small coefficient of compressibility. A pressure up to about 3-5,000 atm. is obtained by this method.

Another way is to utilize the phenomenon of enlarging the volume of some bodies in solidification traction. Everyone knows that bottles filled with water will break if set outside during severe frosts. However, it may

be a definite curiosity that when water is enclosed in a hermetic thick-walled vessel, pressure can be obtained up to 3,000 atm. by this method.

Sometimes methods are used which are based on the rise of pressure during chemical reactions conducted in closed pressure vessels (e.g., in the processes of burning, electrolysis, and the like).

The pressure of a column of liquid is also used and the equation $p = \gamma \cdot gh$ is known to all. For a single column of liquid, using mercury, to attain pressure of 1,000 atm. the vessel would have to be set at a height of 760 m, which, nevertheless, is a fairly difficult matter. This problem can be avoided by using the principle shown in Fig. 2 and using two liquids of different densities. Pressure on the exit of such a system presents the equation: $p = hg(\gamma_2 - \gamma_1)n$, where n is the number of capillaries filled with liquid having the greater density. This method serves to produce very exact model pressures in a range up to 5,000 atm. with the height of the vessels (high pressure capillaries) being up to 100 m.

However, most often pressure is generated in a cylinder with a piston, on which a great force acts, gotten either by hydraulic press, or also great non-transmitted or transmitted load, through a mechanical lever. Limitations of maximum pressure in a piston-cylinder assembly arise from

the finished tear strength of the material, from which the cylinder is made (called the high-pressure chamber), and also from the finished compressive strength of the piston.

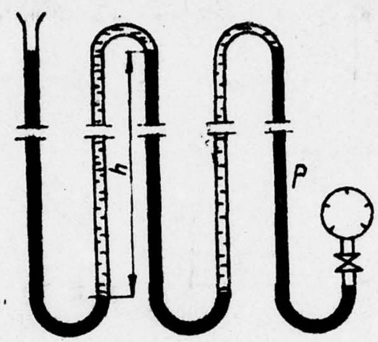


Fig. 2. Production of high pressure in a multiple mercuric column (proj. of Mendeleev). Liquid in black--mercury, with marks--oil.

Single-layer thick-walled cylinders can operate in a range of up to 10-12,000 atm., and in the case of special pressure treatment, depending on the slow overload of the chamber connected with the plastic deformation of its interior, even up to 20,000 atm. Such chambers are made of high quality alloy steels, in special cases (e.g., magnetic or low temperature studies) of beryllium bronze or austentitic steels. These materials are used, of course, for accurate heat and plastic treatment for the purpose of attaining proper parameters of endurance.

A high-pressure piston should have somewhat different properties. Under the influence of vertical pressure it cannot undergo an upset, which would lead to sticking in the cylinder. For that reason also, materials collect on the piston which have a great hardness, first of all, some highly hardened kinds of steel, sintered carbides, and even diamond pistons whenever very high pressure is used.

In practice single-layer cylinders are rarely used, and that is exclusively for pressure under 10,000 atm. With respect to safety as well as prolonging the usefulness of the cylinder, usually multi-layer cylinders are used. Cylinders with such a chamber (2 or more) are pressed one into another in order to produce in the interior part initial compressive stresses. They compensate for tensile stress, which arises in the chamber during the generation of high pressure. Such a solution makes it possible to attain pressures up to 35,000 atm.

It is easy to conclude that in order to maintain high hydrostatic pressures the certainty of a proper seal of the apparatus, in which they are produced, is indispensable.

Elaboration of the seal of the high-pressure piston, making it possible to achieve pressures greater than 10,000 atm., was the result of the

work of the famous American physicist, Nobel Prize winner, P. W. Bridgman (1882-1961). The concept of high-pressure seals (called the principle of uncompensated surface) which he worked out at the beginning of the 20th century is used successfully to this day, and despite the enormous development of high-pressure technology there is no other competitive method.

The principle of the uncompensated surface can best be explained by the example of the so-called Bridgmen type seal piston-cylinder system, shown in Fig. 3. Pressure p, acting on the frontal part of the piston G (so-called high-pressure head) presses down on it by force $F = ps$ to the seal U. This force, acting on the surface s_2 of the seal, produces in it stress, corresponding to a considerably higher pressure than that in the chamber. Thus the pressure of the liquid, approaching the seal by a narrow slit between the chamber wall and the piston, cannot overcome the stress existing in the chamber and get to the outside.

Usually seals of this type are multi-layer, and individual layers are made of materials having different plastic properties. In this case, a few fundamental rules are necessary:

1. The number of rings comprising the individual layers is odd.

2. The principle of the symmetry of changes with regard to the ring placed inside the layer is fulfilled.

3. The interior ring is made of the most plastic material, while the rings placed on both sides are gradually harder.

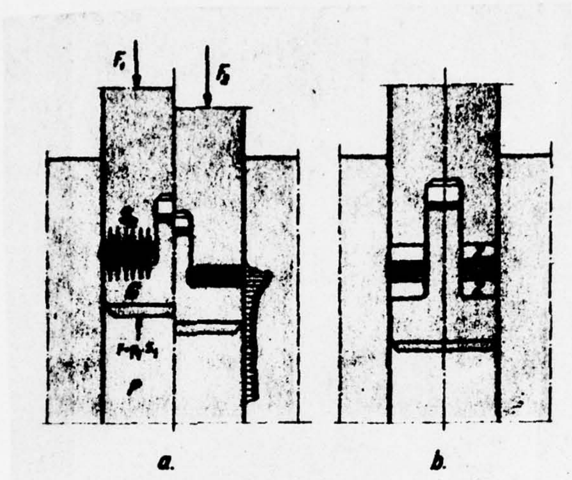


Fig. 3. Bridgman type seal: a--principle of uncompensated surface, arrows indicating the stress distribution in the cylinder walls; b--practically used solution: w--interior seal (teflon), z--exterior seal (copper).

In this way the interior layer of the seal undergoes the greatest strain and it also plays the largest part at low pressures. During this time the somewhat less strained neighboring layers prevent the plastic outflow between the little walls of the piston and the cylinder, of material, from the interior layer. They take over, one by one, the role of actual seals during the increase of pressure.

Another example of a seal based on the principle of the uncompensated surface, is shown in Fig. 4. In this instance, the seal U is produced most often from bronze or soft steel. The ratio of non-compensation, called the surface ratio, on which the pressure acts, to the surface, on which the seal is dependent, is, in the initial phase of the action of the system, very great. Comparatively, for this reason, small pressures lead to the rise on the edge of the seal, of stresses crossing the yield stress of the

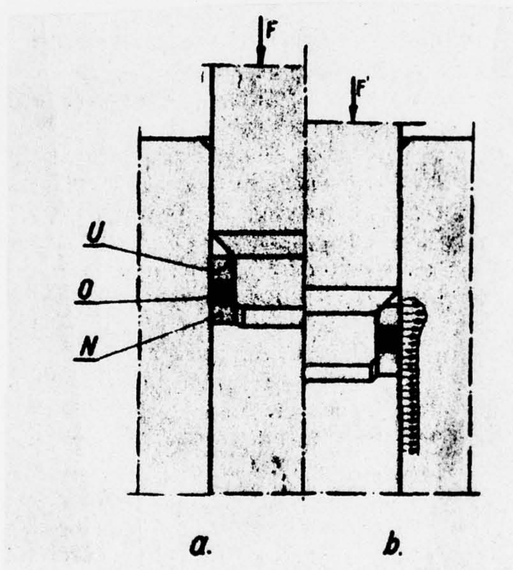


Fig. 4. Seal with uncompensated surface, supported by conicoid surface: a--initial phase of pressure exertion; b--actual seal operation under high pressure.

material from which it is made. As a result the seal distorts in such a way that the surface of the pressure rises to the conic section of the high-pressure piston. Thus the ratio of non-compensation descends until the stresses in the seal become equal, and even lower than the yield stress. During the increase of pressure the process starts anew, during which the seal fills, more and more, the conic space between the chamber and the piston.

The seal system described requires very precise matching of the interior edge of the ring and its lateral surface, to the elements with which it collaborates, the so-called chamber and piston. One of its certain flaws is that it may not seal the high-pressure system in the initial phase, under pressures to the order of several hundreds atm. For that reason this system collaborates usually with preliminary sealing, produced in the form of a rubber or teflon ring of the type O, pressed down initially by nut N. This preliminary seal operates up to the moment when 2-5,000 atm. of pressure is attained, i.e., to that moment when a great certainty exists of the functioning of an actual high pressure seal. Another disadvantage of the system described is that it can be used only once; the deformed seal is not suitable for further use.

There exist two possibilities to seal the piston-cylinder system. The fitst is the seals of the piston, already discussed indetail, which together

with the piston move along the chamber walls during increase or decrease of the pressure. The second possibility is the connection of the seals with the chamber, and not with the piston. They then bear the name of gland seals.

To this time we have explained how pressure can be maintained in a piston-cylinder system, and noted the problem of how pressure can be produced there. This can be effected in many ways: a piston-cylinder system collaborates generally with a hydraulic press or it can also be an integral part of an intensifier. Both these arrangements operate on the well-known principle of hydraulic transmission, which Fig. 5 illustrates. The difference between them lies in the fact that in the presses the quantity obtained at the exit is a large force of pressure (pressure of gun on large surface), while in the intensifiers--high pressure. In the case of hydraulic presses the force used can be not only to work on the high-pressure piston, but to execute industrial operations such as pressing, drawing, and crushing. The pressure ratio along the low- and high-pressure side is the same in both systems, or inversely proportionate to the ratio of the surface of the sections of the low- and high-pressure piston.

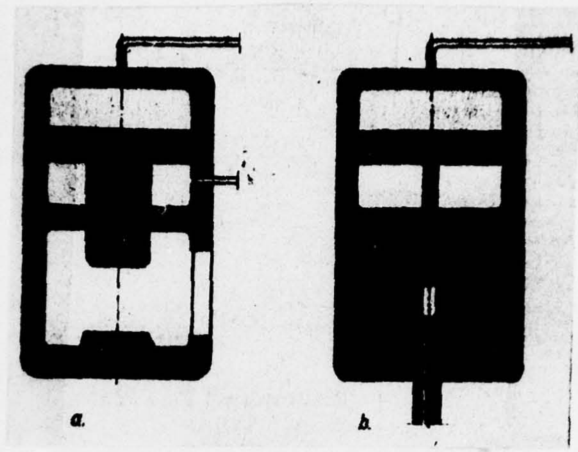


Fig. 5. Simplified principle of action of hydraulic press and high-pressure intensifier: a--press, b--intensifier.

Intensifiers most often collaborate with other high pressure systems (different types with chambers, manostats, and the like) to which the medium transporting the pressure is led by high-pressure capillaries, or special tubes with a high ratio of exterior diameter to interior. This technique is used most often for pressures to the order of 20,000 atm., that is, in the range sufficient for the needs of industrial technology (Fig. 6).

A somewhat different technique is used in the case of the so-called quasi-hydrostatic pressures (almost hydrostatic). This term is used to define that state of compressed material in which small deviations from Pascal's law begin to appear. In other words, the force compressing a body in direction x does not evoke the rise of the forces of reaction in directions y and z having the same value in calculating for a unit of surface. Such an effect appears in solid bodies, until the yield stress is crossed. Upwards

of this stress solid bodies undergo plastic strain and in this state the interior stress distribution is close to being homogeneous, just as takes place in liquids.

Fig. 6. High-pressure hydraulic press with press force of 10,000 T from the Institute of Very High Pressure, Acad. of Sci., U.S.S.R.

These facts make it possible to build high-pressure reaction vessels according to somewhat other principles. In the desire to produce very high pressures, compressed material is placed in a container made of soft metal (e.g., of lead), which then evenly flows around the sample after locking the whole in a high-pressure apparatus. These devices do not have to be sealed so perfectly, as is the case with liquids or gases. Most often the classical cylindrical chamber is given up, which, from the viewpoint of endurance, worked with difficulty to 40-60,000 atm.

From among the structural solutions used in the pressure range of up to 100,000 atm. the most known device is that called the Bridgman anvils. In this device, composed of 2 pistons having a characteristic shape (Fig. 7), the compressed material has the initial form of a thin roller of small height. Such anvils are consequently placed between the piston rods of the hydraulic press having great pressure. Under the influence of this pressure the material begins to flow out to the sides, because of the lack of side sealing. It does not, however, flow out entirely, but the pressure in the interior parts of the roller decreases. The "pressure" distribution which has originated in this way (but actually a unit of surface pressure--see broken line in Fig 7a, connecting the ends of the arrows) is strongly heterogeneous, and the range of highest pressure is very small, even in proportion to the volume of the sample piece. We get a better effect if the sample is placed

inside a sealed rolled ring of such selected properties and dimensions so that its edges would flow out a little like plastic to the outside (Fig. 7b).

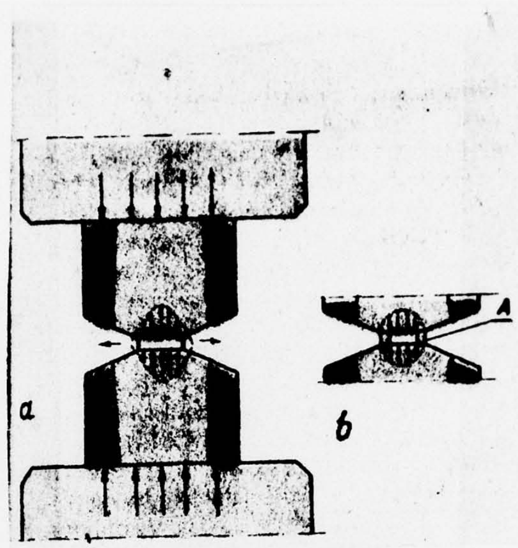


Fig. 7. Bridgman's anvil: a--cross-section of anvils together with sample piece placed between them. Arrows indicate surface pressures; b--distribution of unit of surface pressure in material placed on interior of ring and compressed by anvils.

Bridgman's anvils are made of very hard materials that have great compressive strength (most often they are tungsten sintered carbides). As was already noted, in an apparatus of this type pressure up to 100,000 atm. is achieved, and even several hundred thousands atm. It should be stressed, however, that the volume of the range of high pressure is too small here for this technique to be found useful in industry. In recent years miniature and modified versions of Bridgman's anvils, usually made of one of the

hardest materials, diamond, have been used in scientific investigations. It was by this very technique that there was a successful transition of hydrogen into a metallic state at a pressure of approximately 1.5 mln. atm. As a note of curiosity, it is worth adding that the entire pressure of the anvils did not exceed in this case 30 kG. Thus the readers, themselves, can without difficulty, assess the active surface of such anvils.

For industrial or semi-industrial purposes, and particularly for the synthesis of artificial diamonds, somewhat different equipment is constructed to produce quasi-hydrostatic pressures in relatively large volumes (several cm^3).

The most known among them is the apparatus built by H. T. Hall, comprised of four anvils somewhat similar to Bridgman's anvils. This equipment, having the same principle of operation as described earlier, requires however, the use of special 4-piston rod presses with a spatial arrangement of the piston rods as shown in Fig. 8 (tetrahedrite apparatus). Its advantage is that the material is crushed from four sides, not from two. The maximum volume between tetrahedrite anvils can approach a dozen or so cm^3, This volume is sufficient to conduct semi-industrial synthesis of artificial diamonds (80-100,000 atm. + 3,000 ^oC) or also of Borazon (a variant of boron nitride more resistant to grinding than diamond).

Fig. 8. Tetrahedrite apparatus used to produce quasi-hydrostatic pressures, manufactured in the Institute of Very High Pressures, Acad. of Sci., U.S.S.R.

The 4-piston rod press can be replaced by a system comprised of a standard single-piston press and 3 anvils sliding along the interior conical surface of a large chamber lined with polytetrafluoroethylene foil. The fourth anvil, placed vertically, is pressed by the piston rod of the press and transfers pressure to the remaining anvils and also leads to their displacement to the bottom with the simultaneous concentrated compressing movement by the tetrahedrite piece of material at a given pressure. A serious problem here is the selection of the proper draft angle of the cone,

so that all the anvils have identical feed, and what follows after, an identical pressure.

Modifications of the described apparatus are the solutions including 6 compressing anvils with a cube (Fig. 9). Another structure currently being often used in the process of the synthesis of diamods and Borazon is the so-called Belt type apparatus, also built by H. T. Hall. This type of device, shown in Fig. 10, is actually the union of the modified Bridgman anvils with a high-pressure cylindrical chamber. It is very essential that the interior profile of the chamber be of parabolic shape, which effects a considerable allowance in stress distribution in relation to a plain cylindrical chamber. The inside layer of the chamber is made of sintered carbides and is subject to very great preliminary compressive thrusts, exerted through the exterior ring. Belt type device sets for production of artificial diamonds are mass produced currently in many countries.

In conclusion, it is worth saying a few words about the somewhat different technique of producing high pressures. To this point we wrote of hydrostatic and quasi-hydrostatic pressures. In contradistinction to the situations in which high pressure maintains constant value for a long time, high-pressure processes exist in which the pressure reaches its maximum value for only for a very short time (e.g., for 1/1,000 s or 1/1,000,000 s).

Such pressures are called dynamic. The scope of dynamic pressures produced artificially is very large and currently reaches even several million atm.

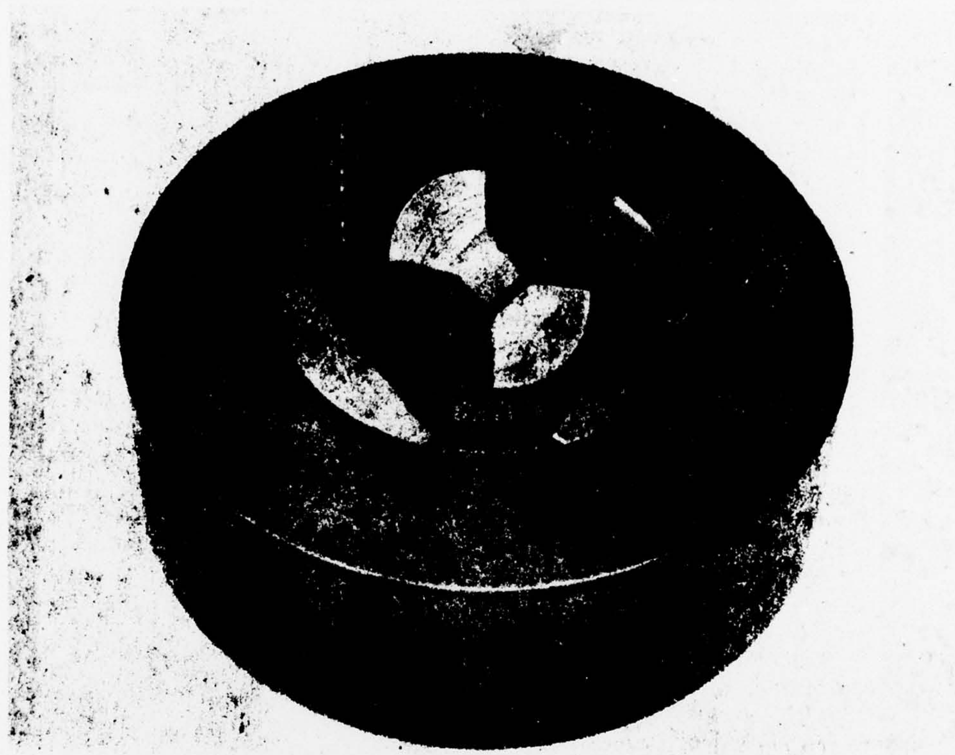


Fig. 9. Photograph of 6-piston rod anvil apparatus manufactured at Physics Institute of Warsaw Polytechnic.

The technology of dynamic pressures depends primarily on the production of local compression of matters in a given medium (in gas, liquid

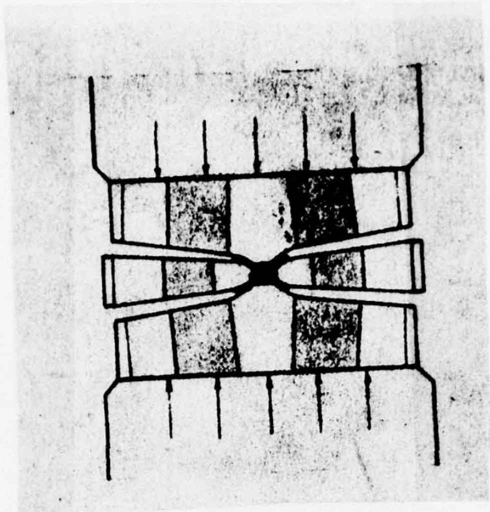


Fig. 10. Belt type apparatus.

or solid state) propagating in the form of a so-called shock wave.

Most often dynamic pressures are produced by explosion and in this form they find industrial application more and more (e.g., for pressure moulding of metals, production of complex squeezed joints, and the like). This technology is well developed in our country, and the "Young Engineer" No. 8, 1974, presented the successes of the Polish scholars from the Army Technical Academy.

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